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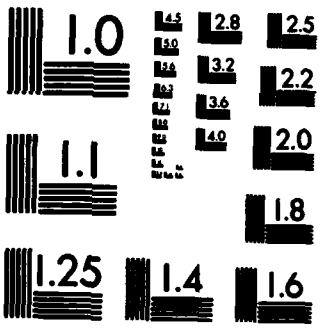

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

An FTIR spectrometer and a thermal analysis system composed of differential scanning calorimeter, thermogravimetric balance, and data station are in use for the study of species adsorbed to carbon black and of polymer networks.

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INSTRUMENTS FOR CHARACTERIZATION OF CARBON BLACK AND POLYMERS

FINAL REPORT

Warren T. Ford and Mark G. Rockley

September 12, 1984

U.S. Army Research Office

Contract No. DAAG-83-G-0099

Oklahoma State University
Department of Chemistry
Stillwater, OK 74078

Content of Final Report

Contract DAAG29-83-G-0099 was awarded under the DoD University Research Instrumentation Program solely for the acquisition of instruments. The instruments support primarily onging ARO research programs under contracts

DAAG29-82-K-0133, Macroporous Polymeric Supports for Organic Synthesis, Warren T. Ford, Principal Investigator

DAAG-29-81-K0098

Mark G. Rockely, Principal Investigator

Detailed technical reports based on research with the instruments will be provided in the interim and final techncial reports on those contracts. In this final report we list the instruments acquired, brief statements of the problems studied, research in progress, and most important results. No funds in this instrumentation contract supported personnel.

Instruments Purchased

1. Thermal Analysis System: Perkin-Elmer Corp. model DSC-2C differential scanning calorimeter, model TGS-2 thermogravimetric balance, and model TADS data station with accessories for DSC experiments at subambient temperatures.

2. Fourier Transform Infrared Spectrometer: IBM Instruments Inc., IR-32, 1 cm^{-1} resolution. Associated Pronto dual 5-Megabyte drive computer, Model 16/200.

Summary of Results to Date (September 1, 1984).

1. Thermal Analysis

An investigation of glass transitions in crosslinked polystyrene gels is in progress. The aim is to relate glass transitions and any higher temprature transitions that can be detected to the behavior of solvent-swollen crosslinked polystyrenes as they are used in polymer-supported synthesis and as they are analyzed by higher resolution and magic angle spinning solid state NMR spectroscopy. Key questions to be answered are: Can synthetic reagents be transported in and out of polystyrene networks at or below the glass transition temperature (T_g) at rates fast enough to perform synthetic chemistry? If transport is adequately fast, are chemical reactions in the network at $< T_g$ markedly retarded compared with reactions at $> T_g$? Does the dramatic broadening of high resolution ^{13}C NMR spectra observed at 1.2-1.5 x T_g in polymer melts extend to polymer gels?

At present nothing is known about thermal transitions in highly swollen polymer networks. Previous studies of polymer/solvent systems best described as plasticized polymers have been carried out to < 30 wt % solvents. Our aim is to determine T_g in networks containing up to 80 wt % solvent. Several

empirical equations have been used to relate T_g of plasticized polymers to T_g of the polymer and T_g of the plasticizer (solvent). We aim to test which, if any, of these equations best describes T_g of highly swollen networks.

The experimental work in progress employs a series of polystyrenes crosslinked with 1 to 10% divinylbenzene and swollen with "good" solvents. T_g is determined by differential scanning calorimetry in the range 100 K to 400 K, depending on the solvent. The high sensitivity of the thermogravimetric balance is required to weigh precisely the polymer/solvent composition of each sample. The calorimetric measurements are carried out with sealed smaples to prevent solvent evaporation and under inert atmosphere at subambient temperatures to prevent condensation of atmospheric moisture. Efforts so far have been devoted to perfection of the experimental methods. Answers to the fundamental questions about T_g's in polymer gels are not yet available.

Fourier Transform Infrared Spectroscopy

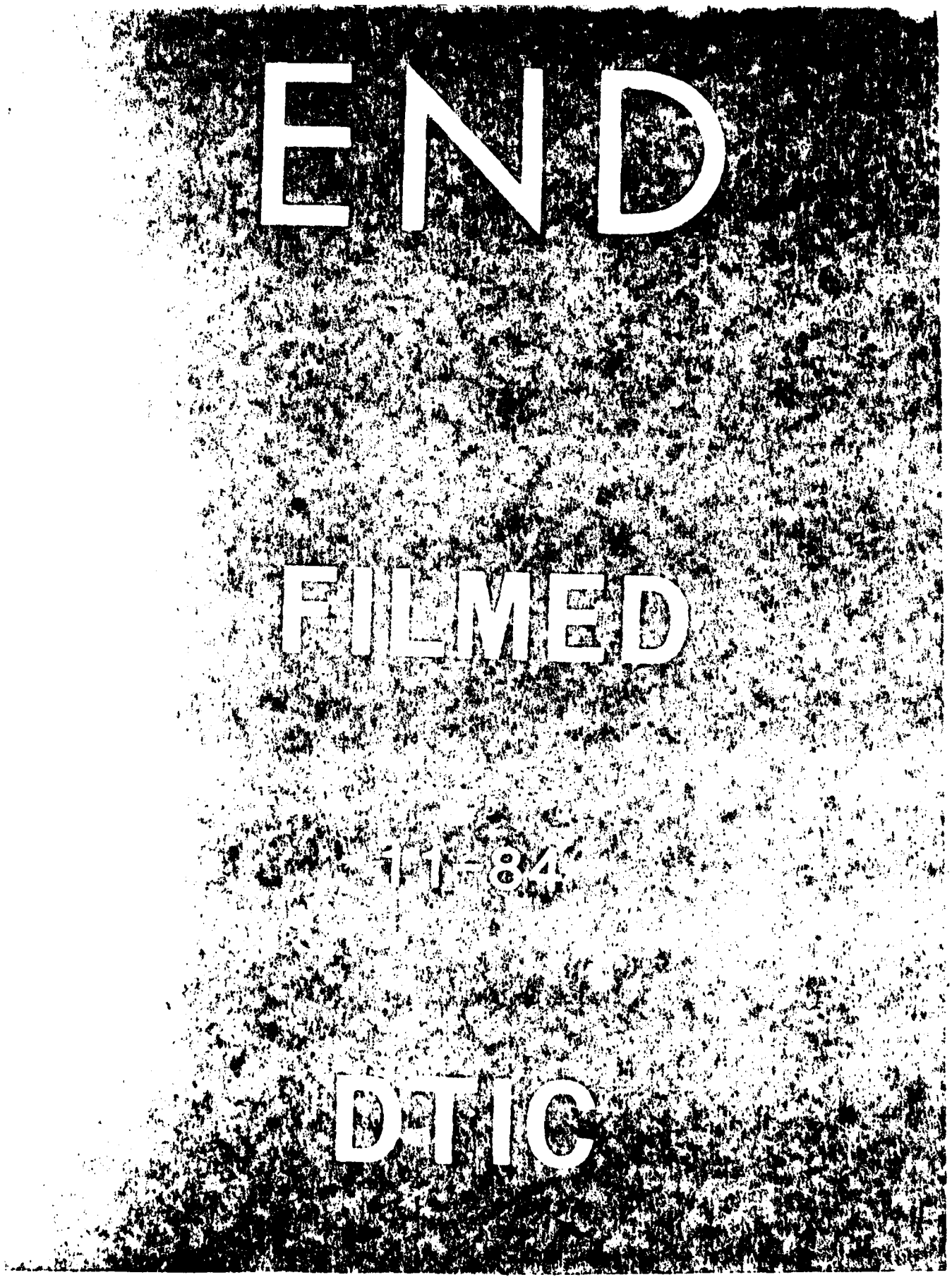
The FTIR was originally purchased to free up time on the main Digilab FTS-20C spectrometer. That purpose was achieved. In addition it has proved invaluable in obtaining thin film transmission spectra of carbon-black on salt windows (with and without sorbates). Two manuscripts resulting from these analyses are currently being prepared: one involves DMMP simulant adsorption; the other involves aromatic and aliphatic alcohol adsorption.

The new FTIR also is in use for analysis of the polymer-supported reagents used in synthesis under contract DAAG29-83-K-0133.

Publications

Two manuscripts on FTIR of carbon black are in preparation and will be forwarded upon completion.

Additional publications on polymer networks resulting from research with the thermal analysis and FTIR instruments will be provided as soon as they are prepared.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